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A NEW TETRA-EPOXIDE RESIN AS A MATRIX FOR ADVANCED COMPOSITES.(U)

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A NEW TETRA-EPOXIDE RESIN AS A MATRIX FOR ADVANCED COMPOSITES

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SUMMARY

Two versions of a new tetra-epoxide resin were supplied by Shell Laboratories, for evaluation as a possible matrix for carbon fibre.

Because of the absence of tack in the resulting pre-preg both resins were modified at RAE with a minor percentage of the Bisphenol A resin, Shell 828.

All four formulations (*ie* modified and unmodified 114 and 115 resins) had their curing characteristics established and were then used in conjunction with Courtaulds XAS carbon fibre, the reinforcement being in the form of satin-weave and unidirectional fabrics.

The mechanical properties of the derived laminates were obtained at room temperature, at 80°C and 130°C. Resistance to a range of aircraft fluids was also examined.

The best results overall were obtained with the lower molecular weight epoxide blended with Bisphenol A resin; and it is recommended that more extensive trials and long-term testing should begin.

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1 INTRODUCTION

As part of the RAE programme on the development and evaluation of potential matrix resins for composite materials, an extra-mural contract was placed with the Shell Chemical Company in 1979.

The objective was to seek inexpensive alternatives to the epoxide compound tetraglycidyl-4,4'-diaminodiphenyl methane (which is the basis for many of the resin systems presently used by the aircraft industry¹) without substantial sacrifice either of ease of processing, or of mechanical properties, of the resulting laminates.

Also of importance were the resistance to the range of normal aircraft fluids and the amount of water uptake at saturation, which has an effect on compressive performance under hot, wet conditions².

After preliminary synthetic work at Shell, a new tetra-epoxide was selected as a suitable candidate; and two versions, in a higher and lower molecular weight were submitted to Materials Department for examination. The resins are designated as Shell 1153/114 for the low molecular weight and Shell 1153/115 for the higher molecular weight materials*. Both are pale yellow, brittle solids as received, freely soluble in acetone, methyl ethyl ketone, etc.

Stoichiometric amounts of diaminodiphenyl sulphone, which is well-known as a heat-resistant aromatic amine, were used as the hardener, with a small addition of boron trifluoride etherate as catalyst.

The reinforcement of particular interest is carbon fibre. It was used in the form of standard unidirectional and satin-weave fabrics, based on Courtaulds XAS grade for which data are available with other resin systems³.

In the reconnaissance reported here, the curing and moulding behaviour of the Shell resins, both unmodified and modified, is reported, together with the mechanical properties of the derived laminates.

2 MATERIALS USED

2.1 Resins and hardeners

The two experimental tetra-epoxide resins, designated 114 and 115, were supplied by Shell Chemicals as solid castings which could be easily broken up into small pieces convenient for dissolving in acetone. The lower molecular weight resin 114, has an epoxide equivalent weight of 212, whilst that of the 114 system is 235. Melting points of the two resins are 38-43°C for the 114 resin and 64-69°C for the 115 material.

In addition to the two principal resins used, the well known DGEBA resin Epikote 828 supplied by Shell Chemicals, was used as a modifier.

The curing agent used throughout the programme was diaminodiphenyl sulphone (DDS) supplied by Koch-Light Laboratories Limited. Shell Chemicals Epikure BF₃400, boron trifluoride monoethylamine complex, was used as a catalyst.

* These will be called 114 and 115 resins throughout the text.

2.2 Carbon fibre reinforcements

Two carbon fibre fabrics were chosen, a five-shaft satin and a tied unidirectional type. Both cloths were based on Courtauld's 3000 filament XAS carbon fibre and were woven by Carr Reinforcements Limited. The unidirectional material was held together with an 11 tex glass fibre weft; this being preferred as the result of a previous investigation⁴.

3 MANUFACTURE OF PRE-PREG

Solutions of the resins were prepared by dissolving the appropriate amount of resin and hardener in acetone so as to give a 40% w/w solution. The amount of DDS curing agent required was slightly different for each resin, this being dependent on the epoxide content.

The carbon fibre fabrics were impregnated with the solutions by brush coating. After being allowed to stand for 3 h the pre-preg was placed in an air-circulating oven, set at 100°C, for 20 min. The pre-pregs produced had a resin solids content of 40% by weight.

The 114 and 115 resins, being solids at room temperature, were found to produce pre-pregs with little or no tack.

In an attempt to improve the tack and flexibility of the pre-pregs produced, increasing proportions of the liquid DGEBA resin Epikote 828 were added to the prime resin. It was found that a 33% by weight addition was needed to give a satisfactory pre-preg.

The detailed formulations used for the modified and straight resins are given below:

| 114 resin | 114-828 blend | 115 resin | 115-828 blend |
|-------------------------|---------------------------|-------------------------|---------------------------|
| 100 g 114 | 100 g 114 | 100 g 115 | 100 g 115 |
| 30 g DDS | 50 g 828 | 27 g DDS | 50 g 828 |
| 1 g BF ₃ 400 | 47 g DDS | 1 g BF ₃ 400 | 42 g DDS |
| | 1.5 g BF ₃ 400 | | 1.5 g BF ₃ 400 |

4 USE OF TORSIONAL BRAID ANALYSIS (TBA) TO FOLLOW CURE OF RESINS

In order to establish the optimum cure conditions for the resins under evaluation, the Polymer Chemistry Section, Materials Department, RAE, carried out a series of isothermal runs to determine the gel and vitrification times at various temperatures. The TBA results are presented in Figs 1 and 2. It is clear from these results that the higher molecular weight resin, 115 cures more rapidly than the 114 system and that the addition of the Epikote 828 resin slows down the cure of both the 114 and 115 systems. The TBA work also showed that if the modified resins were post-cured at a temperature of 190°C, they both achieved a glass transition temperature (T_g) of between 241 and 245°C after 12 h. It was found however that the T_g had reached about 95% of this value after only 2 h post cure.

5 PREPARATION OF LAMINATES

All the laminates were compression moulded in a light-alloy tool of internal dimensions 315 mm x 215 mm. The moulding tool was coated with Vydex release agent prior to use.

The moulding procedure was the same for both the satin-weave and unidirectional fabric and is outlined below.

The pre-preg materials were cut to size and the requisite number of layers placed in the cold mould; this being eight for the satin-weave and 15 for the unidirectional fabric. Each layer of satin-weave pre-preg was stacked in the mould with the warp fibres along the 0° axis.

The loaded mould was placed in an electrically heated hydraulic press, the temperature of the platens being maintained at 150°C. The moulding was allowed to dwell for some minutes with only contact pressure being applied; this time varied between 12 min for the 115 system, and 18 min in the case of the 114-828 formulation. After the dwell period the mould was pressed down to 1.9 mm stops in the case of the satin-weave fabric and 2.0 mm for the unidirectional fabric, a pressure of approximately 2 MPa being required for this.

The mould was left in the press for a further 1 h before turning the heaters off and allowing it to cool. Once the temperature had dropped below 60°C the mould was removed from the press, and the laminate ejected from it.

All the laminates were subjected to a post-cure. The schedule followed was, 2 h at 170°C plus 16 h at 190°C in the case of the 114 based laminates, and 16 h at 190°C for 115 based laminates.

6 TESTING PROCEDURES

Determinations of flexural strength, tensile strength, tensile modulus and interlaminar shear strength were all performed at room temperature, and in addition, the flexural and interlaminar shear strengths were determined at 80°C and 130°C. The test specimens were cut from moulded sheets using a diamond-edged cutting wheel (152 mm diameter x 1.6 mm thickness), the grit size being 85-100. The material was tested in the longitudinal (warp) direction on a floor-mounted Instron machine (type 1115). Ten replicate specimens were normally taken for each test. The details of the various mechanical tests performed are given below.

(i) Flexural strength

A standard specimen, 100 mm x 10 mm, as described by Sturgeon⁵ was used.

The specimen was loaded at a rate of 20 mm/min.

(ii) Tensile modulus

A parallel sided specimen 200 mm x 10 mm was used. Light-alloy end-pieces were bonded to the ends of each specimen using Redux 410 epoxy adhesive.

The specimens were loaded at a rate of 2 mm/min to about 50% of their expected failing load and the strain measured using an Instron strain gauge extensometer.

(iii) Tensile strength

Once the tensile modulus of the satin-weave specimens had been determined they were tested to failure. The specimens made from the unidirectional fabric had a radius ground on them in accordance with the recommendations of Dootson⁶, before testing to destruction.

The load was applied at a rate of 2 mm/min.

(iv) Interlaminar shear strength

A standard specimen 12 mm x 10 mm, as described by Dootson⁶ was used.

The rate of loading was 2 mm/min.

7 RESULTS OF MECHANICAL TESTS

The detailed room temperature properties are given in Tables 1 and 2, whilst Tables 3 and 4 show the effect of increasing temperature on the flexural and interlaminar shear strengths.

Considering the room temperature results first, both the 114 and 115 systems performed quite well. It is clear however, that the 114 systems are somewhat superior to those based on 115. The addition of the Epikote 828 resin to both 114 and 115, showed no adverse effect; indeed an *increase* in properties took place in most cases. This general increase in the mechanical properties with the addition of Epikote 828 may be associated with better adhesion of the matrix to the carbon fibre; the interlaminar shear values being higher in every case.

At 80°C the flexural strength of all the resin/fabric combinations held up well; 90% or more retention in all but one case. It is evident from both the flexural and interlaminar shear strengths at 80°C that the addition of Epikote 828 has given improved retention of properties. Little difference between the 114 and 115 resins was noted with regard to the percentage retention of properties.

When the temperature was increased to 130°C the flexural strength of the systems containing the 828 addition again showed improved retention of strength as compared with the base systems. The 114-828 resin reinforced with satin-weave fabric retained an excellent 93% of the room temperature value, although the same resin reinforced with the unidirectional material, retained only 73%. A similar difference between the satin-weave and the unidirectional fabric was found with the 114 system. It is interesting to note that in a previous evaluation of another resin system⁷, the unidirectional fabric was found to perform somewhat worse at elevated temperatures than, in that case, an unwoven unidirectional carbon fibre. It would appear that the presence of a small amount of glass weft may adversely affect the strength retention of CFRP at elevated temperatures.

The interlaminar shear values followed the same trends as those observed with flexural strength except that the reduction in properties was generally rather more marked. However, despite the reduction of values, the results are still quite acceptable even at 130°C; 56 MPa for example in the case of unidirectional fabric with the 114-828 matrix.

8 RESISTANCE TO AIRCRAFT FLUIDS

A small exercise was performed to see whether some of the commonly encountered aviation fluids were likely to have any serious effect on carbon fibre composite material, containing the 114-828 resin system.

Eight different fluids were selected, mostly chosen from British Standard 3G.100:

Part 2: sub-section 3.12, and these are listed in Table 5. In addition to the eight aircraft fluids used, the effect of water was also assessed.

Small pieces of unidirectional composite 100 mm × 10 mm × 2 mm in size, were carefully weighed and then immersed in the various fluids which were kept at a temperature of 50°C. After 48 h the pieces of composite were removed from the fluid and the weight change recorded. The specimens were returned to the fluids and then removed and re-weighed after a total time of 7 days. Before the specimens could dry-out, their flexural strengths were also measured and these results are given in Table 5 together with the recorded weight changes.

These show that for most of the fluids tested, there was a negligible change in weight and an unchanged flexural strength as compared with the control. The greatest change was produced by water, which gave a weight pick-up of about 0.5% after 7 days immersion at 50°C accompanied by a 14.3% reduction in flexural strength.

9 DISCUSSION AND CONCLUSIONS

In assessing the value of a new resin system we are interested first in the absolute level of mechanical properties at room temperature as compared with the same reinforcement in standard matrices; and second in the reduction in mechanical properties with elevated temperature.

In the case of XAS fibre, information is available from previous work with the Ciba-Geigy XD 927 resin^{3,4}, with the Friedel-Crafts system Xylok 237⁷ and with the Shell pre-condensate system Epikote DX 210^{7,8}. This information is summarised for unidirectional material at room temperature:

| Matrix | Flexural strength (MPa) | Tensile strength (MPa) | Interlaminar shear strength (MPa) |
|---|----------------------------|---------------------------|---|
| 114-828 | 2018 | 1480 | 92 |
| XD 927 | 2027 | 1840 | 73 |
| DX 210/BF ₃ 400 (unwoven) | 1919 | 1567 | 99 |

For satin-weave fabrics, laminates tested at room temperature gave:

| Matrix | Flexural strength (MPa) | Tensile strength (MPa) | Interlaminar shear strength (MPa) |
|---------|----------------------------|---------------------------|---|
| 114-828 | 918 | 783 | 51 |
| XD 927 | 1020 | 791 | 58 |

The extent of reduction in properties, as the temperature is raised from room temperature, through 80°C to 130°C is given by:

| Matrix/fibre | Temperature | Flexural strength (% retention) |
|--------------|-------------|------------------------------------|
| 114-828/XAS | RT | 100 |
| | 80°C | 91 |
| | 130°C | 73 |
| DX 210/AS | RT | 100 |
| | 80°C | 88 |
| | 120°C | 76 |

These results taken together show that the new resin system gives very similar room temperature figures to other resins with both kinds of woven reinforcement. At elevated temperatures, strength retention is rather better than for a Friedel-Crafts system⁷, comparing favourably with Epikote DX 210.

The following points may be made in conclusion:

- (1) Of the two versions of the tetra-epoxide supplied, the lower molecular-weight version, resin 114, is more convenient to use and gives better mechanical properties at room temperature and above.
- (2) The blend of 114 with 828 resin is better than the unmodified 114 alone, in respect of both tack and flexibility in the pre-preg form.
- (3) The 114-828 system is easy to handle, has a convenient cure and post-cure schedule and gives a final Tg in the region of 240-245°C.
- (4) The strength and stiffness of laminates at room temperature is high, with good strength retention at elevated temperatures. Depending on the end use, an upper working limit of 130°C is possible.
- (5) There is good resistance to the normal range of aircraft fluids, with an acceptably low moisture absorption.
- (6) The 114-828 blend seems to be a very good general purpose pre-preg system for carbon fibre and it is recommended that full-scale trials be mounted both for pre-pregging on commercial equipment and for the necessary long-term testing in fatigue, heat-ageing, weathering and moisture resistance.

Acknowledgments

The torsional braid analysis results were provided by Dr W.A. Lee and Mr M.J. Oliver, using the special automatic-recording TBA apparatus developed in the Polymer Chemistry Section, Materials Department.

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Table 1

ROOM TEMPERATURE PROPERTIES OF LAMINATES MADE WITH THE VARIOUS
RESIN SYSTEMS AND SATIN-WEAVE CLOTH

| Resin | Flexural strength (MPa) | Tensile strength (MPa) | Tensile modulus (GPa) | Interlaminar shear strength (MPa) |
|---------|--------------------------|--------------------------|------------------------|-----------------------------------|
| 114 | Mean = 881 CV = 6.2% | Mean = 743 CV = 8.1% | Mean = 68 CV = 8.9% | Mean = 47 CV = 7.8% |
| 114-828 | Mean = 918 CV = 9.7% | Mean = 783 CV = 13.2% | Mean = 73 CV = 4.6% | Mean = 51 CV = 9.9% |
| 115 | Mean = 822 CV = 11.2% | Mean = 691 CV = 7.9% | Mean = 68 CV = 7.3% | Mean = 42 CV = 6.8% |
| 115-828 | Mean = 859 CV = 8.5% | Mean = 802 CV = 4.9% | Mean = 71 CV = 3.5% | Mean = 53 CV = 13.5% |

$V_f = 0.60$, CV = coefficient of variation

Table 2

ROOM TEMPERATURE PROPERTIES OF LAMINATES MADE WITH 114 AND 114-828
RESIN SYSTEMS WITH UNIDIRECTIONAL CLOTH

| Resin | Flexural strength (MPa) | Tensile strength (MPa) | Tensile modulus (GPa) | Interlaminar shear strength (MPa) |
|---------|--------------------------|--------------------------|-------------------------|-----------------------------------|
| 114 | Mean = 2150 CV = 3.5% | Mean = 1631 CV = 7.8% | Mean = 131 CV = 2.6% | Mean = 88 CV = 11.1% |
| 114-828 | Mean = 2018 CV = 8.2% | Mean = 1480 CV = 5.5% | Mean = 131 CV = 4.3% | Mean = 92 CV = 5.2% |

$V_f = 0.62$

Table 3

THE EFFECT OF INCREASING TEMPERATURE ON THE FLEXURAL STRENGTH OF LAMINATES
MADE WITH 114 OR 115 BASED RESIN

| Temperature of test $^{\circ}\text{C}$ | 114 Satin-weave | 114-828 Satin-weave | 115 Satin-weave | 115-828 Satin-weave | 114 Unidirectional | 114-828 Unidirectional |
|--|------------------------------|-----------------------------|------------------------------|-----------------------------|------------------------------|------------------------------|
| 23 | Mean = 881 CV = 6.2% | Mean = 918 CV = 9.7% | Mean = 822 CV = 11.2% | Mean = 859 CV = 8.5% | Mean = 2150 CV = 3.5% | Mean = 2018 CV = 8.2% |
| 80 | Mean = 822(93) CV = 9.2% | Mean = 888(97) CV = 9.3% | Mean = 778(95) CV = 12.7% | Mean = 829(97) CV = 9.5% | Mean = 1786(83) CV = 7.5% | Mean = 1840(91) CV = 6.3% |
| 130 | Mean = 752(85) CV = 11.5% | Mean = 850(93) CV = 6.6% | Mean = 686(83) CV = 14.3 | Mean = 715(83) CV = 8.6% | Mean = 1390(65) CV = 6.5% | Mean = 1474(73) CV = 5.3% |

Units - MPa, () = % retention of room temperature strength, CV = coefficient of variation

Table 4

THE EFFECT OF INCREASING TEMPERATURE ON THE INTERLAMINAR SHEAR STRENGTH
OF LAMINATES MADE WITH 114 OR 115 BASED RESIN

| Temperature of test $^{\circ}\text{C}$ | 114 Satin-weave | 114-828 Satin-weave | 115 Satin-weave | 115-828 Satin-weave | 114 Unidirectional | 114-828 Unidirectional |
|--|----------------------------|----------------------------|----------------------------|-----------------------------|----------------------------|----------------------------|
| 23 | Mean = 47 CV = 7.8% | Mean = 51 CV = 9.9% | Mean = 42 CV = 6.8% | Mean = 53 CV = 13.3% | Mean = 88 CV = 11.1% | Mean = 92 CV = 5.8% |
| 80 | Mean = 44(94) CV = 4.2% | Mean = 47(92) CV = 7.3% | Mean = 36(86) CV = 6.4% | Mean = 39(74) CV = 8.0% | Mean = 65(74) CV = 4.1% | Mean = 71(77) CV = 5.3% |
| 130 | Mean = 40(85) CV = 6.0% | Mean = 43(84) CV = 6.6% | Mean = 35(83) CV = 6.3% | Mean = 34(64) CV = 17.5% | Mean = 50(57) CV = 3.9% | Mean = 56(61) CV = 3.7% |

Units - MPa, () = % retention of room temperature strength

Table 5

EFFECT OF IMMERSION IN VARIOUS AIRCRAFT FLUIDS AT 50°C ON
114-828 UNIDIRECTIONAL COMPOSITE

| Fluid | % Weight change 48 h | % Weight change 7 days | Flexural strength* (MPa) 7 days |
|---|-------------------------|---------------------------|------------------------------------|
| Methyl ethyl ketone | -0.116 | -0.044 | 2166 |
| Avtur 50 | -0.076 | -0.044 | 2070 |
| Paint stripper (methylene chloride based) | +0.171 | +0.281 | 2064 |
| Castrol OM 15 (mineral hydraulic oil) | -0.087 | -0.079 | 2081 |
| Skydrol B (phosphate ester based hydraulic oil) | -0.078 | -0.016 | 2227 |
| OX 38 (synthetic lubricating oil) | -0.029 | -0.037 | 2037 |
| Kilfrost ABC (Propylene glycol based de-icer) | +0.143 | +0.434 | 1926 |
| Genklene (1,1,1-trichloroethane) | -0.126 | -0.028 | 2120 |
| Water | +0.169 | +0.462 | 1730 |

* Initial flexural strength = 2018 MPa

Fig 1

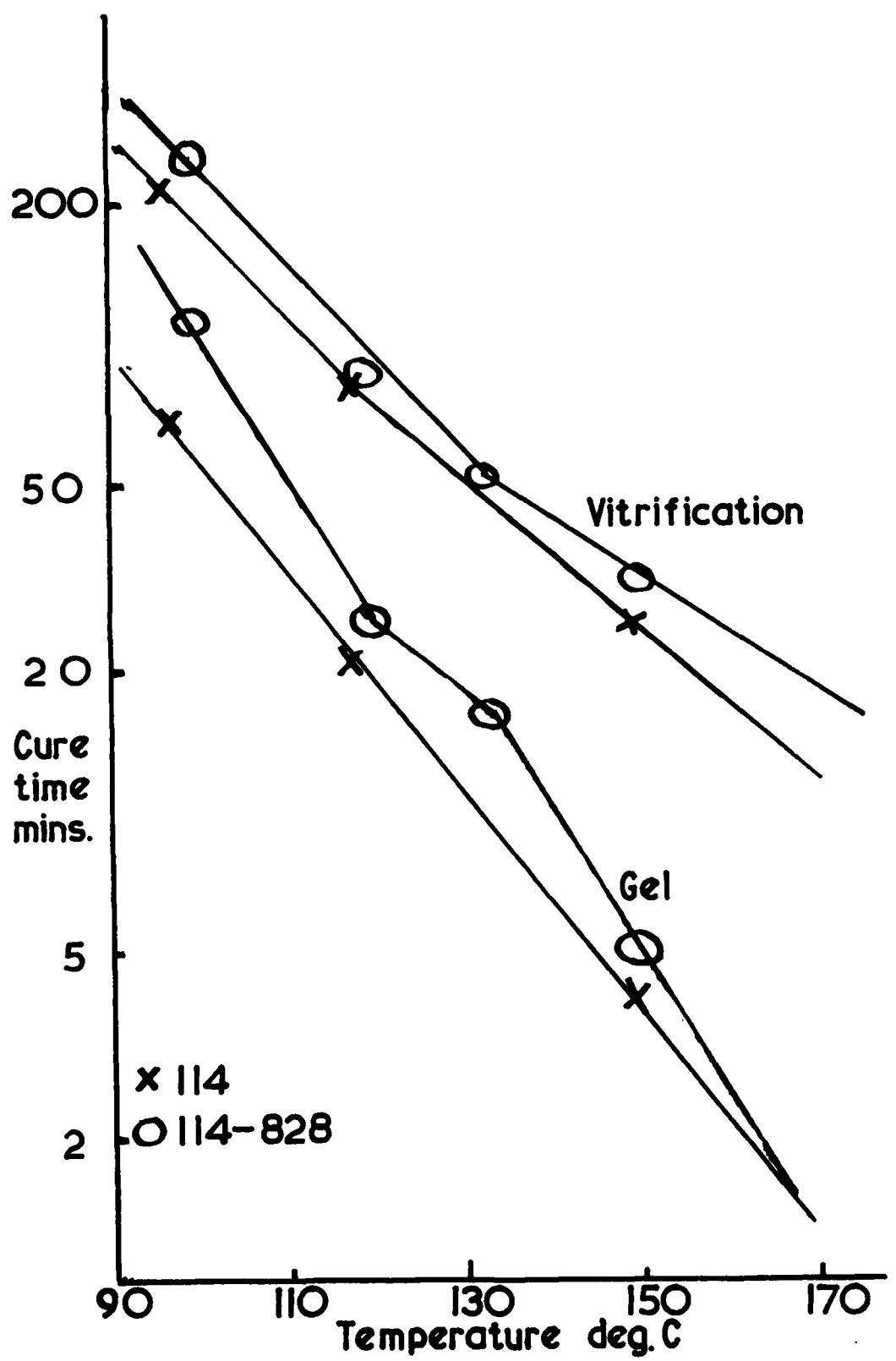


Fig 1 Effect of temperature on the gel and vitrification times of 114 and 114-828 resin systems

Fig 2

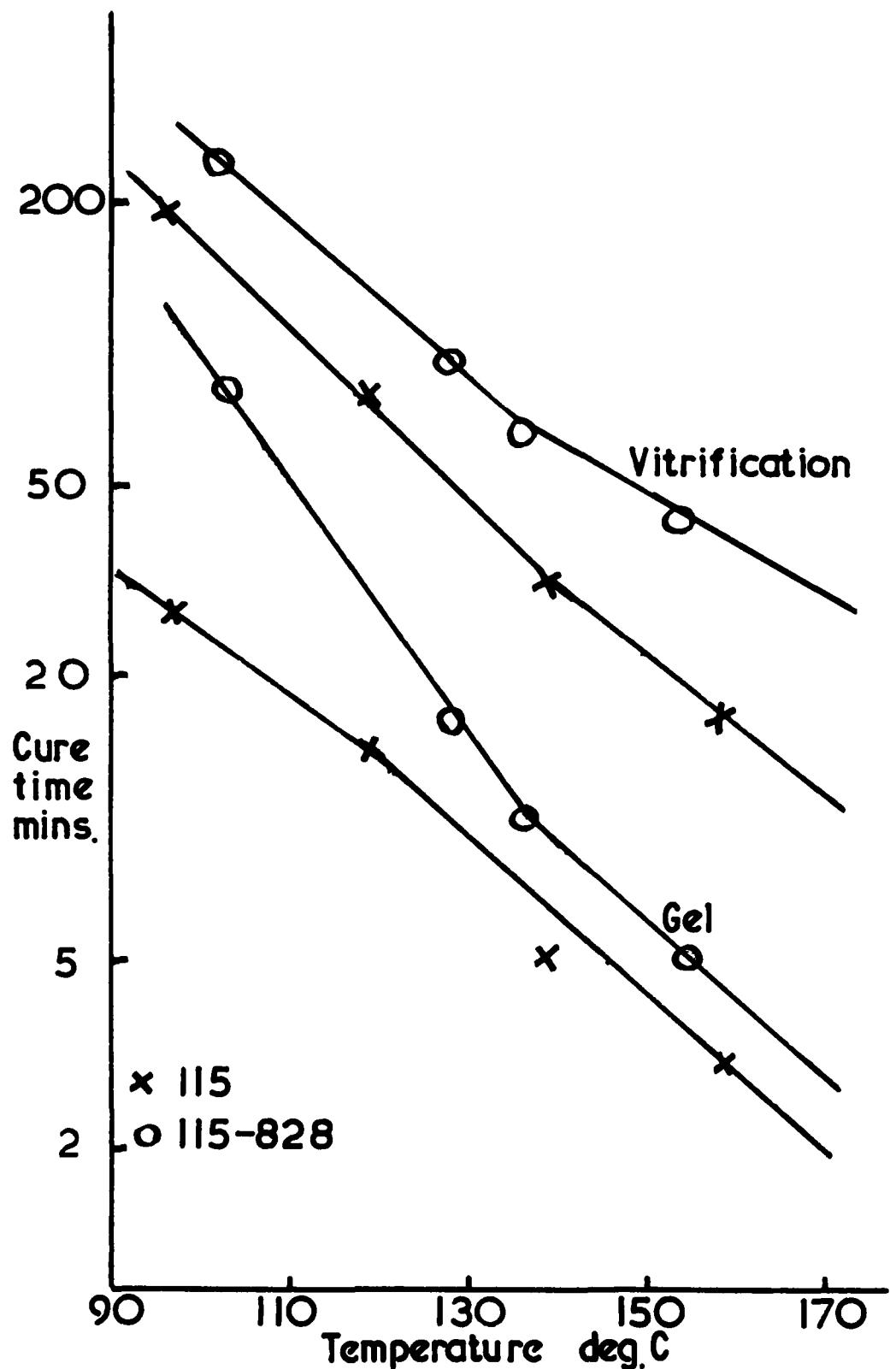


Fig 2 Effect of temperature on the gel and vitrification times of 115 and 115-828 resin systems

REPORT DOCUMENTATION PAGE

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| 17. Abstract Two versions of a new tetra-epoxide resin were supplied by Shell Laboratories, for evaluation as a possible matrix for carbon fibre. Because of the absence of tack in the resulting pre-preg both resins were modified at RAE with a minor percentage of the Bisphenol A resin, Shell 828. All four formulations (i.e modified and unmodified 114 and 115 resins) had their curing characteristics established and were then used in conjunction with Courtaulds XAS carbon fibre, the reinforcement being in the form of satin-weave and unidirectional fabrics. The mechanical properties of the derived laminates were obtained at room temperature, at 80°C and 130°C. Resistance to a range of aircraft fluids was also examined. The best results overall were obtained with the lower molecular weight epoxide blended with Bisphenol A resin; and it is recommended that more extensive trials and long-term testing should begin. | | | |

